

ARIZONA DEPARTMENT OF TRANSPORTATION

REPORT NUMBER: FHWA/AZ-82/159/SUMMARY

CHEMICAL AND PHYSICAL PROPERTIES OF ASPHALT- RUBBER MIXTURES — PHASE III

VOLUME — SUMMARY

Prepared by:

J.C. Rosner, Ph.D., P.E.
J.G. Chehovits, P.E.

June 1982

Final Report

Prepared for:

U.S. Department of Transportation
Federal Highway Administration
Arizona Division

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle Chemical and Physical Properties of Asphalt-Rubber Mixtures - Phase III, Summary Report		5. Report Date April, 1982	
		6. Performing Organization Code 2390J030	
7. Author(s) J. C. Rosner and J. G. Chehovits		8. Performing Organization Report No.	
9. Performing Organization Name and Address Western Technologies, Inc. 3737 East Broadway Road Phoenix, Arizona 85036		10. Work Unit No.	
		11. Contract or Grant No. HPR-1-19(159)	
12. Sponsoring Agency Name and Address Arizona Department of Transportation 206 South 17th Avenue Phoenix, Arizona 85007		13. Type of Report and Period Covered Final	
		14. Sponsoring Agency Code	
15. Supplementary Notes In cooperation with the U. S. Department of Transportation Federal Highway Administration			
16. Abstract An extensive statistically designed laboratory investigation which had objectives of evaluating (1) effects of asphalt-rubber components on mixture properties, (2) effects of diluent on mixture properties, (3) effects of temperature on mixture properties, (4) applicability of several test procedures for asphalt-rubber mixtures, and (5) differences between field and lab produced asphalt-rubber mixtures was performed. Asphalt-rubber materials formulated with six rubber types from two different production processes, four rubber concentrations, four asphalts and four diluent concentrations were produced and tested. Field produced mixtures were obtained from experimental asphalt-rubber field projects in Arizona. Testing procedures included absolute viscosity, Schwyer rheometer, sliding plate microviscometer, force-ductility, and viscosity during mixing. The study concluded that asphalt-rubber mixture properties vary depending on the rubber type, rubber concentration, asphalt type, diluent concentration, diluent cure time, and temperature. Temperature susceptibility of asphalt-rubber mixtures containing rubber from the two production processes studied varied. Mixtures produced in the laboratory were found to be stiffer than field produced mixtures. The force-ductility and sliding plate microviscometer tests were found to yield acceptable testing variability to permit their use in specifying asphalt-rubber physical properties.			
17. Key Words Asphalt-rubber Torque-Fork Force-Ductility Reclaimed rubber Schwyer Rheometer		18. Distribution Statement No restrictions. Available to the public through the NTIS, Springfield, Virginia 22161	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 87	22. Price

CHEMICAL AND PHYSICAL PROPERTIES
OF ASPHALT - RUBBER MIXTURES - PHASE III

SUMMARY REPORT

BY
J. C. ROSNER, J. G. CHEHOVITS

SUBMITTED TO

THE ARIZONA DEPARTMENT OF TRANSPORTATION
HIGHWAY DIVISION
PHOENIX, ARIZONA

FOR

RESEARCH PROJECT - ARIZONA HPR 1-19(159)

SPONSORED BY

THE ARIZONA DEPARTMENT OF TRANSPORTATION
IN COOPERATION WITH
THE U. S. DEPARTMENT OF TRANSPORTATION
FEDERAL HIGHWAY ADMINISTRATION

WESTERN TECHNOLOGIES, INC.
3737 EAST BROADWAY ROAD
PHOENIX, ARIZONA

APRIL, 1982

ACKNOWLEDGEMENTS

Western Technologies, Inc. wishes to express its appreciation to the Research Division of Arizona Department of Transportation and the U. S. Department of Transportation, Federal Highway Administration for funding and administration of the study.

The writers wish to thank Mr. Gene R. Morris, Engineer of Research, Mr. Frank M. McCullagh, Senior Research Engineer and Mr. Joe DeVito, Project Coordinator of the Research Division of Arizona Department of Transportation for their fine cooperation during this project.

The assistance of Sahuaro Petroleum and Asphalt Company, and Arizona Refining Company during the project is sincerely appreciated.

Finally, the dedicated work of many individuals at Western Technologies, Inc. in laboratory testing, data collection, data reduction and clerical work is appreciated.

PROJECT PERSONNEL

Western Technologies, Inc.

1. J. C. Rosner: Principal Investigator
2. R. D. Pavlovich: Co-Principal Investigator
3. J. G. Chehovits: Secondary Investigator
4. P. C. Stubblebine: Data Analyst
5. P. F. Feliz: Technician
6. T. V. Morris: Technician
7. R. R. Bradley: Research Chemist
8. D. Paluszcyk: Technician
9. J. P. McCarthy: Technician
10. T. D. Carr: Chemist
11. C. Appleby: Draftsman
12. K. L. Blake: Typist

Petroleum Sciences, Incorporated

1. R. L. Dunning, Consultant
2. J. E. Johnson, Technician

Arizona Department of Transportation

1. G. R. Morris: Research Manager
2. F. M. McCullagh: Senior Research Engineer
3. J. A. DeVito: Project Coordinator

INDEX

	<u>PAGE NO.</u>
TECHNICAL REPORT DOCUMENTATION PAGE	i
ACKNOWLEDGEMENTS	ii
PROJECT PERSONNEL	iii
INDEX	iv
1.0 PROJECT DESCRIPTION.....	1
2.0 ASPHALT-RUBBER AS A PAVING MAINTENANCE MATERIAL.	3
3.0 MATERIALS.....	8
4.0 EXPERIMENTAL DESIGNS AND DATA ANALYSIS.....	20
5.0 ASPHALT-RUBBER MIXTURE PREPARATAION.....	21
6.0 TEST PROCEDURES.....	22
7.0 EFFECTS OF RUBBER TYPE, CONCENTRATION AND ASPHALT.....	40
8.0 EFFECTS OF ASPHALT.....	46
9.0 EFFECT OF DILUENT.....	50
10.0 EFFECT OF TEMPERATURE.....	54
11.0 PHYSICAL PROPERTIES OF FIELD-MIXED ASPHALT-RUBBERS.....	58
12.0 COMPARISON OF LAB AND FIELD-MIXED ASPHALT-RUBBERS.....	63
13.0 CONCLUSIONS.....	69
REFERENCES.....	71
APPENDIX A	73
APPENDIX B	82

1.0 PROJECT DESCRIPTION

1.1 The objectives of this project were to:

- Evaluate the effects of the components of asphalt-rubber mixtures on physical properties of the mixtures,
- Evaluate the effects of temperature on physical properties of asphalt-rubber mixtures,
- Determine physical properties of field-produced asphalt-rubber mixtures,
- Compare properties of field and lab produced asphalt-rubber mixtures, and
- Evaluate the feasibility of using testing procedures employed in this investigation for asphalt-rubber specification purposes.

1.2 The above objectives were accomplished by performing and analyzing results of several series of statistically designed laboratory experiments utilizing different asphalt-rubber mixtures.

1.3 The experiments performed considered asphalt-rubber mixtures formulated with:

- Six different types of rubber from two different production processes,
- Four different rubber concentrations,
- Four different asphalts, and
- Four different concentrations of diluent and five curing times.

1.4 Field produced mixtures which were characterized in the lab were obtained from an asphalt-rubber experimental project constructed in Arizona from October to December of 1978.

1.5 Material properties assessed in this study were:

- Absolute viscosity at 140F (60C)

- Apparent viscosity and shear rate sensitivity by the Schwyer rheometer at -20, 39.2, and 77F
 - Stress, strain, and creep compliance properties at -20, 39.2, and 77F using force-ductility.
 - Apparent viscosity, creep, strain recovery, rebound and rheological characteristics at 32F (0C) using the sliding plate microviscometer
 - Ring-and-Ball Softening Point
 - Fracture Temperature
 - Viscosity during mixing at 375F (191C) using the Arizona Torque-Fork
 - Viscosity during mixing at 375F (191C) using the Haake Rotational Viscometer.
- 1.6 Analytical techniques used included conventional one, two, three, and four-way analyses of variance, a means ranking procedure, and graphical plots.
- 1.7 Details of the experiments, results, and conclusions are reported in five separate volumes of the report, "Chemical and Physical Properties of Asphalt-Rubber Mixtures - Phase III" as follows:

Volume I	Effects of Rubber Type, Concentration, and Asphalt
Volume II	Effects of Asphalt
Volume III	Effects of Diluent
Volume IV	Physical Properties of Field-Mixed Asphalt-Rubber Mixtures and Com- parison of Lab and Field-Mixed Asphalt-Rubbers
Volume V	Effects of Temperature

2.0 ASPHALT-RUBBER AS A PAVING MAINTENANCE MATERIAL

2.1 Introduction

Asphalt-rubber can be defined as a mixture of asphalt cement, granulated reclaimed tire rubber, and possibly certain additives. Asphalt-rubber is distinguished from a material generally termed rubberized asphalt in that asphalt-rubber contains a higher amount of rubber (15 to 25% by weight) than rubberized asphalt (1 to 3% by weight).

2.1.1 In recent years, asphalt-rubber materials have proven useful in pavement maintenance in several functional applications including:

- Asphalt concrete binders
- Surface treatments
- Stress absorbing membranes for reduction of reflective cracking
- Waterproofing membranes
- Crack and joint sealants

2.1.2 Presently, the major emphasis for usage of asphalt-rubber materials is in pavement maintenance applications - specifically, for use in reducing reflective cracking. Use of asphalt-rubber as a crack and joint sealer is becoming increasingly more popular.

2.1.3 The asphalt-rubber concept was developed by Charles H. McDonald while he was Engineering Supervisor for the City of Phoenix, Arizona in the early 1960's (1). The material was used mainly as a surface patching material for cracked pavement sections.

2.1.4 Since the mid 1960's, performance of asphalt-rubber materials has been studied in numerous field test projects. The major functional uses studied have been as stress absorbing membrane interlayers for prevention and control of reflective cracking and as seal coats.

2.2 Reaction Mechanism

2.2.1 When rubber and asphalt cement are mixed at high temperatures (250-400F), the rubber particles swell to several times their original volume (2). Swelling is postulated to occur due to physical and chemical interactions between rubber particles and asphalt. Swelling results in an increase in viscosity of the asphalt-rubber mixture, and is commonly referred to as the reaction between asphalt and rubber.

2.2.2 The reacted asphalt-rubber material has radically differing properties from the original asphalt cement and also from the unreacted asphalt-rubber material. Properly reacted asphalt-rubber materials have sufficient stress and strain tolerance such that the materials can bridge underlying pavement cracks and withstand deformations without fracturing. Additionally, properly reacted asphalt-rubber is extremely sticky having excellent adhesion to aggregate and pavement surfaces which is very important in paving applications.

2.3 Component Materials

Mixture properties of asphalt-rubber are influenced by the properties and characteristics, both physical and chemical, of component materials.

2.3.1 The specific asphalt cement used in asphalt-rubber will not only influence the viscosity of the mixture due to asphalt grade and consistency, but, its chemical compositional characteristics may influence the degree and rate of swelling of rubber particles thus influencing properties of the reacted asphalt-rubber material.

2.3.2 Rubber characteristics including particle size and shape, surface texture, rubber source and composition, and rubber concentration in the mixture have been noted to influence the properties of asphalt-rubber mixtures (3, 4). Particle size, shape and surface texture, and rubber source and composition can influence the degree and rate of swelling or reaction which occurs between rubber and asphalt. Rubber concentration in the mixture can influence the mass properties of reacted asphalt-rubber mixtures (3).

- 2.3.3 Diluents are sometimes added to commercial asphalt-rubber mixtures to reduce viscosity of the reacted product so that it can be sprayed through a distributor truck. It has been noted that diluents tend to inhibit rubber particle swelling (1).

2.4 Uses of Asphalt-Rubber

Major uses of asphalt-rubber in paving have been in maintenance related operations - chip seals for stress absorbing membranes and hot-poured joint sealers.

- 2.4.1 Chip seals using asphalt-rubber as the binder have been found to perform well as low-modulus stress-absorbing-membranes (SAM) or stress-absorbing-membrane-interlayers (SAMI) for aiding in reduction of reflective cracking. Numerous field test projects using asphalt-rubber in this application have been constructed and are well documented (5, 6, 7, 8, 9). Performance records indicate that asphalt-rubber chip seals used in either a SAM or SAMI functional application can aid in reduction of reflection of fatigue cracks but will not prevent reflection of thermally-induced transverse cracks (10).

- 2.4.2 Effective joint and crack sealers for asphalt and concrete pavements have been made from asphalt-rubber materials (11). Asphalt-rubber crack sealers have the ability to withstand differential crack movement, bond strongly to crack faces, and reheal or rebond to joint faces in warm weather if fracture or separation from crack faces occurs.

2.5 Recent Research Developments

Considerable amounts of research work related to performance of asphalt-rubber mixtures in varying applications has been performed in recent years.

- 2.5.1 Laboratory research related to asphalt-rubber mixtures has primarily been concerned with studying asphalt-rubber reactions, applicability of standard asphalt testing procedures, and unique characteristics of asphalt-rubber materials alone and in combination with aggregates.

- 2.5.1.1 The basic materials properties of asphalt-rubber were studied and reported by Green and Tolonen (1). Properties studied included rubber swelling characteristics in asphalt, viscosity, and strain recovery characteristics. Testing procedures for asphalt-rubber materials are recommended.
- 2.5.1.2 Oliver (3) studied the elastic behavior of asphalt-rubber mixtures using a sliding plate rheometer. It was noted that digestion (reaction) time and temperature can influence the elastic recovery of asphalt-rubber mixtures and that effects were different with different rubber types. It was also concluded that rubber concentration also influences elastic recovery, but particle size does not. Additionally, an asphalt-rubber produced in the laboratory was found to have similar elastic properties to field produced asphalt-rubber composed of the same materials and reacted at the same temperature for the same time period.
- 2.5.1.3 Frobel, Jimenez, and Cluff (4) studied properties of asphalt-rubber mixtures as related to use as a waterproof membrane. Conclusions reached during this study include:
- Permeability of asphalt-rubber materials is so low that they can be considered to be impermeable.
 - Asphalt-rubber mixtures have decreased ductility when compared to that of the base asphalt and that finer rubber particles result in higher ductilities than larger particles.
 - Toughness (resistance to deformation) of an asphalt-rubber mixture increases as the rubber particle size increases.
 - Viscosity of asphalt is greatly increased by the addition of crumb rubber particles.

2.5.1.4 Pavlovich, Shuler and Rosner (12) reported on a study which investigated the effects of reaction time and temperature on a single asphalt-rubber mixture using several modified standard asphalt testing procedures and non-standard procedures.

The study concluded that:

- Modified standard tests studied (softening point, absolute viscosity, and ductility) are not applicable for testing asphalt-rubber without modification of testing method and precision statement.
- Force-ductility testing can detect differences in reaction time and temperature used in preparation of asphalt-rubber mixtures.
- The Schweyer Rheometer can be used to determine low temperature viscosity of asphalt-rubber materials.
- More research work related to testing procedures and asphalt-rubber reactions is required before test methods for specification purposes can be developed.

3.0 MATERIALS

3.1 Rubber. Six different types of granulated rubber were used in the experiments performed in this study. Rubber from two different production processes, ambient grind tread peel crumb and ambient grind high natural rubber content devulcanized crumb, were studied, each at three differing particle size distributions (gradations).

3.1.1 The ambient grind tread peel crumb rubber was produced by Atlos Rubber Reclaiming of Los Angeles, California.

3.1.1.1 The material consisted of ground (crumb) rubber produced by mechanically grinding passenger car tread peel at ambient temperatures. No additional processing or depolymerization was used on the ground rubber product to alter material properties prior to mixing with asphalt. The addition of a maximum of 4 percent calcium carbonate powder prior to packaging is permitted to prevent the rubber particles from sticking together.

3.1.1.2 Two Atlos rubber products which differed in gradation were studied. These products are designated by Atlos as TP044 and TP027. TP044 is composed mostly of rubber particles sized between the #8 (0.0937 in.) and #30 (0.0234 in.) mesh screens, while TP027 is mostly between the #30 (0.0234 in.) and #100 (0.0059 in.) mesh screens. Additionally, in this study, a 50/50 mixture by weight of TP044 and TP027 was studied to investigate effects of a more well-graded ambient grind tread peel crumb rubber product on asphalt rubber mixture properties.

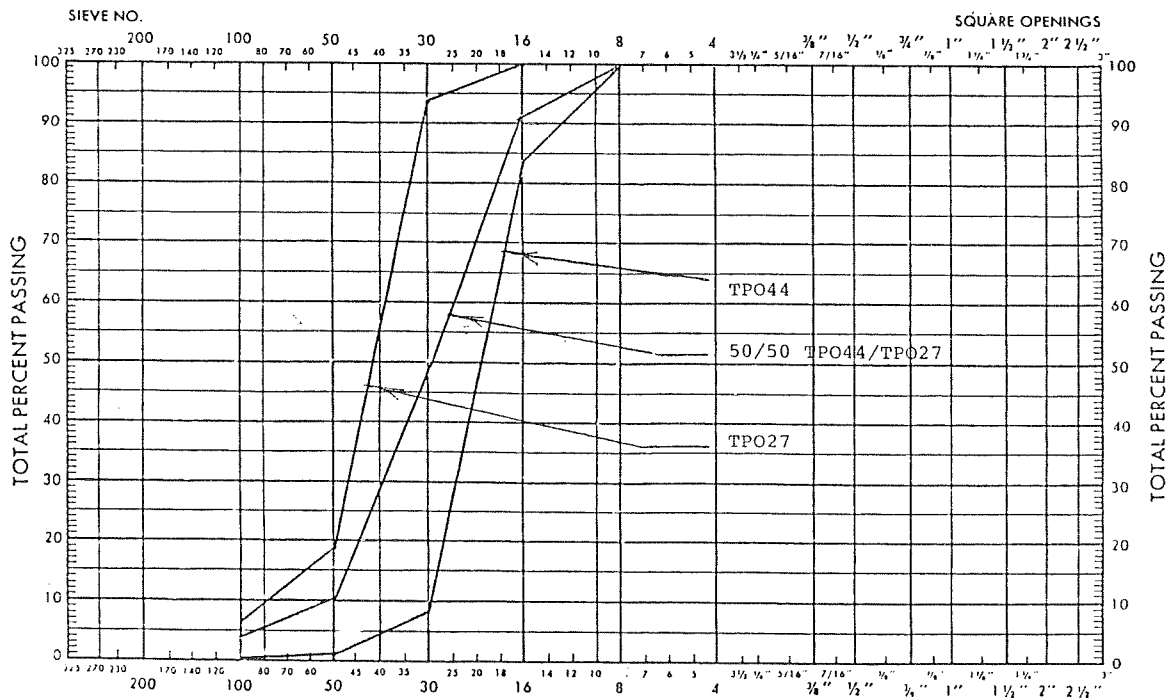
3.1.1.3 Gradations of TP044, TP027, and the 50/50 mixture of TP044 and TP027 are tabulated in Table 1 and plotted in Figure 1.

3.1.2 Devulcanized ambient grind crumb rubber with a high natural rubber content produced by U.S. Rubber Reclaiming of Vicksburg, Mississippi was the second source of rubber studied in this project.

TABLE 1

GRADATIONS OF ATLOS TP044, TP027, AND A 50/50
MIXTURE (by weight) OF TP044 AND TP027

Sieve Size	% Passing		50% TP044 50% TP027
	TP044	TP027	
#8	100	100	100
#16	83	100	91
#30	7	94	50
#50	1	19	10
#100	0.5	6.5	3.5
#200	0.0	0.0	0.0



U.S. STANDARD SIEVES—ASTM DESIGNATION E 11

Figure 1 Gradations of Atlos TP044, TP027, and a 50/50
Mixture (by weight of TP044 and TP027)

3.1.2.1 The material consists of a blend of 40 percent powdered reclaimed (devulcanized) rubber and 60 percent ambient ground vulcanized rubber which contains a high natural rubber content. The rubber is free from fabric, wire, or other contaminants except that up to 4 percent calcium carbonate may be added to prevent caking.

3.1.2.2 Two U.S. Rubber Reclaiming products which differed in gradation were studied. The first is designated as GT274 which is composed mostly of particles sized between the #16 (0.0469 in.) and #100 (0.0059 in.) mesh sieves. The second is a finer material obtained from grinding buffings produced during production of GT274. This material is designated as U.S. Rubber Fine (USRF) and is sized mostly between the #30 (0.0234 in.) and #100 (0.0059 in.) mesh sieves.

Additionally, a 50/50 mixture by weight of GT274 and USRF was studied to investigate effects of a more well-graded high natural rubber content devulcanized rubber product on asphalt-rubber mixture properties.

3.1.3 Each of the six rubber types described above (two production processes each at three gradations) was incorporated into asphalt-rubber mixtures at 15, 20, 25, and 30 percent by weight of total asphalt-rubber mixture.

3.1.4 Scanning electron photomicrographs of TPO44 and GT274 rubber particles are shown in Figure 3 at magnifications of 70 and 250 times. The smaller particle size of GT274 is noted and it can be seen that GT274 appears to have a rougher surface texture than TPO44.

3.2 Asphalt Cement. Four different asphalt cements were used in asphalt-rubber mixtures for this study. Three of the asphalt cements used were a combination of an AR4000 and 3 different percentages of an extender oil. The fourth asphalt used was an AR1000.

TABLE 2

GRADATIONS OF U.S. RUBBER RECLAIMING GT274,
U.S. RUBBER FINES, AND A 50/50 MIXTURE
(by weight) OF GT274 AND U.S. RUBBER FINE

Sieve Size	% Passing		50% GT274 50% USRF	
	GT274	USRF	50%	USRF
#8	100	100	100	
#16	98	100	99	
#30	76	95	86	
#50	23	33	28	
#100	8.0	12.2	10.1	
#200	0.0	0.0	0.0	

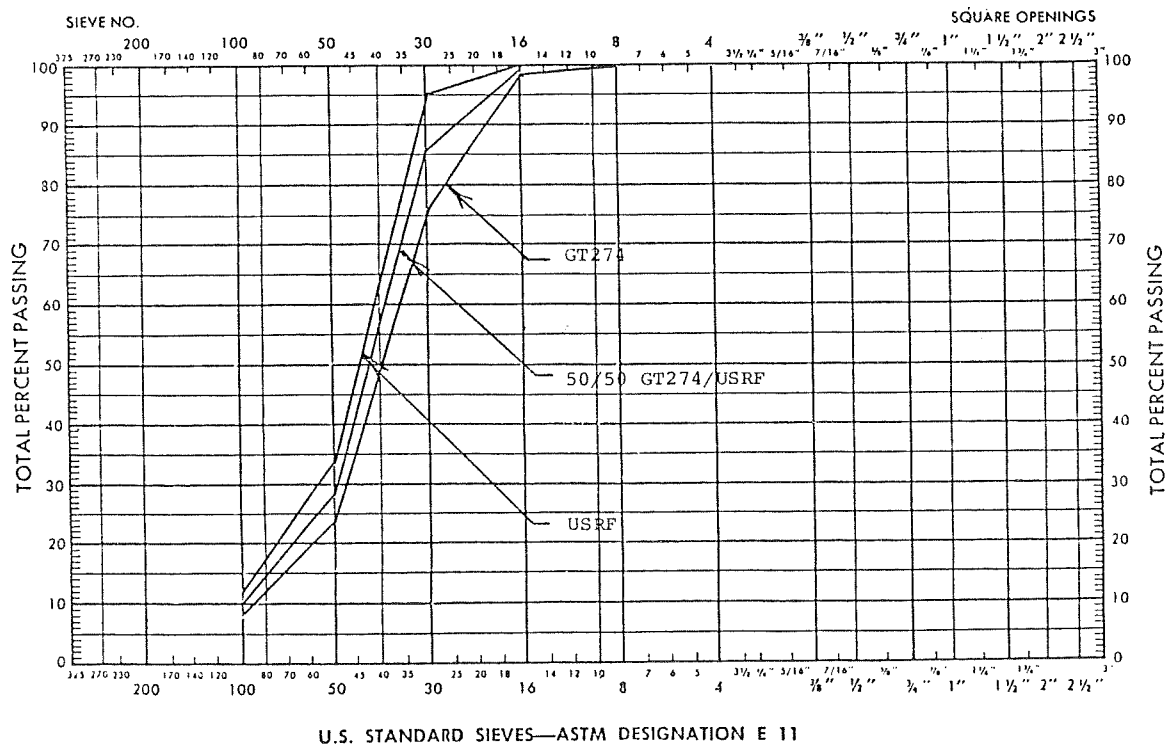
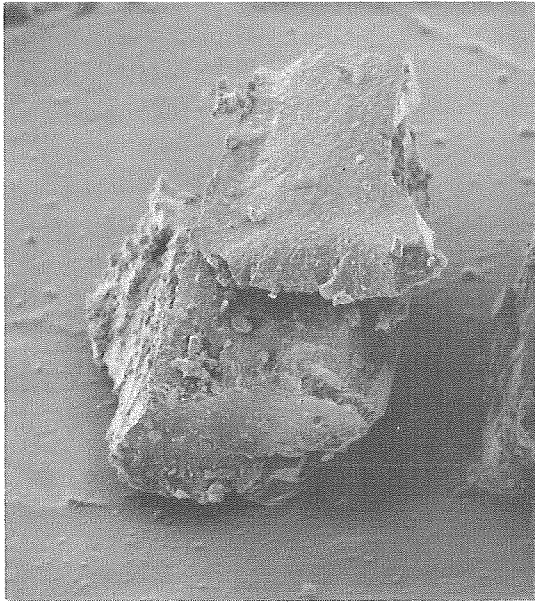
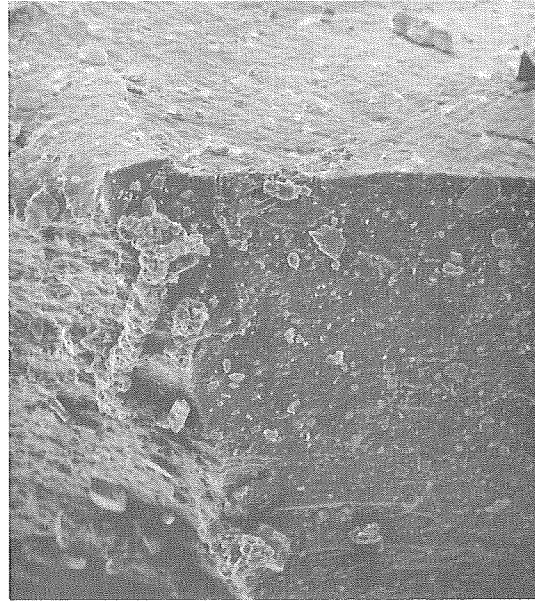


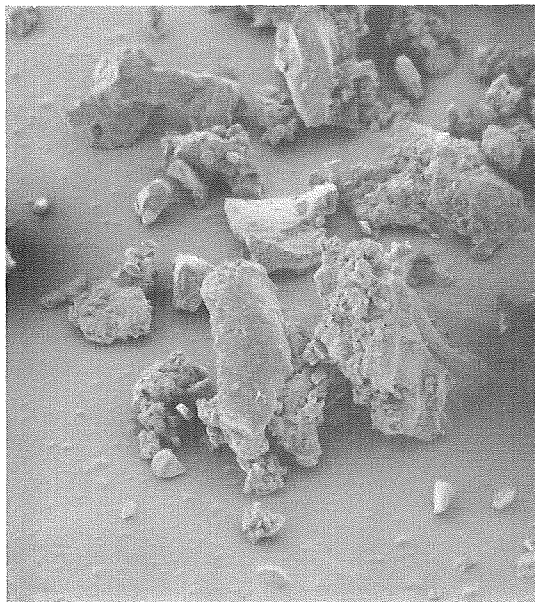
Figure 2 Gradations of U.S. Rubber Reclaiming GT274,
U.S. Rubber Fines, and a 50/50 Mixture
(by weight) of GT274 and U.S. Rubber Fine



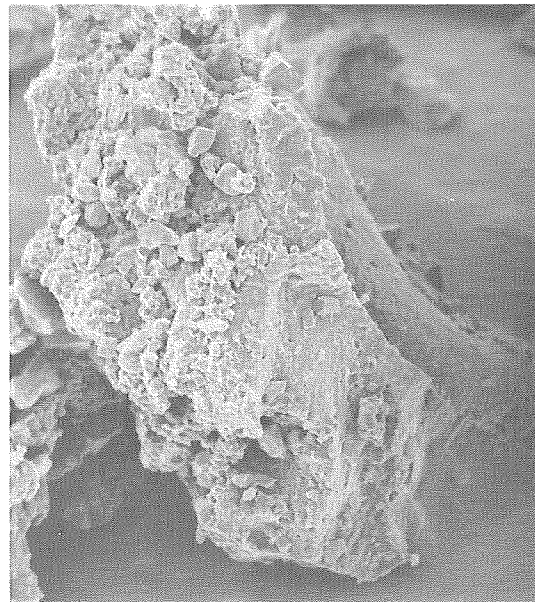
TP044 70X



TP044 250X



GT274 70X



GT274 250X

Figure 3

- 3.2.1 The AR1000 used in this study was supplied by Sahuaro Petroleum and Asphalt Company of Phoenix, Arizona. This asphalt is commonly used in conjunction with approximately 25 percent Atlos TP044 rubber and diluents to produce commercial asphalt-rubber mixtures.
- 3.2.2 The AR4000 used in this study was supplied by Arizona Refining Company (ARCO) of Phoenix, Arizona. This asphalt is commonly used in conjunction with 20 percent U.S. Rubber Reclaiming GT274 rubber and 2 percent extender oil to produce commercial asphalt-rubber mixtures.
- 3.2.3 The extender oil used in this study was Califlux GP manufactured by the Golden Bear Division of Witco Chemical Corporation and supplied by Sahuaro Petroleum and Asphalt Company.
- 3.2.4 Penetration, softening point, absolute viscosity, kinematic viscosity, ductility, flash point and solubility tests were performed for both AR4000 and AR1000 asphalt cements. Test procedures used to characterize asphalts are tabulated in Table 3. Test results and ASTM D3381-76 (13) specification limits for viscosity-graded asphalt cements are tabulated in Tables 4 and 5.
 - 3.2.4.1 The Sahuaro AR1000 meets ASTM D3381-76 requirements for an AR1000 asphalt cement.
 - 3.2.4.2 The ARCO AR4000 meets ASTM D3381-76 requirements for an AR4000 asphalt cement.
- 3.2.5 Penetration, absolute viscosity, and kinematic viscosity tests were performed on the mixtures of unaged AR4000 with Califlux at 2, 4, 8, 15, and 25 percent by weight of asphalt. Test results, along with results for unaged AR4000 and AR1000 asphalts are tabulated in Table 6.
 - 3.2.5.1 Based on test results in Table 5, two percentages of Califlux additions in the AR4000 were studied. A 15 percent addition was selected to produce an asphalt similar in characteristics to the AR1000, and a 6 percent addition was selected to produce a material with characteristics falling between the AR4000 with 2 percent Califlux and the AR4000 with 15 percent Califlux.

TABLE 3

TEST PROCEDURES USED TO
CHARACTERIZE ASPHALT CEMENTS

<u>Property</u>	<u>Testing Procedures</u>
Penetration	ASTM D5-49, "Penetration of Bituminous Materials"
Softening Point	ASTM D36-76, "Softening Point of Bitumen (Ring-and-Ball Apparatus)"
Absolute Viscosity	ASTM D2171-78, "Viscosity of Asphalts by Vacuum Capillary Viscometer"
Kinematic Viscosity	ASTM D2170-76, "Kinematic Viscosity of Asphalts (Bitumens)"
Ductility	ASTM D113-79, "Ductility of Bituminous Materials"
Flash Point	ASTM D92-78, "Flash and Fire Points by Cleveland Open Cup"
Solubility	ASTM D2042-76, "Solubility of Asphalt Materials in Trichlorethylene"
Rolling Thin Film	ASTM D2872-80, "Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin Film Oven Test)"

TABLE 4

PHYSICAL PROPERTIES OF UNAGED SAHUARO
AR1000, AGED RESIDUE AND
ASTM D3381 SPECIFICATION LIMITS

<u>Property</u>	<u>Test Results</u>		<u>Specification Limits</u>
	<u>Unaged</u>	<u>RTFOT¹ Aged Residue</u>	
Penetration, 100g, 5 sec, 77F; 1/10 mm	127	86	65 min ²
Softening Point; °C	41.0	46.0	--
Absolute Viscosity, 60C, 30 cmHg; Poise	662	1062	1000+250 ²
Kinematic Viscosity, 135C; cSt	178	233	140 min ²
Ductility, 77F, 5cm/min; cm	-	100	100 min ²
Flash Point, COC; °F	490	--	400 min
Solubility in Trichlor- ethylene; %	99.95	--	99.0 min
<hr/>			
Rolling Thin Film Oven Loss; %		0.32	

Notes: ¹Rolling Thin Film Oven Test
 ²Tests on RTFOT Residue

TABLE 5

PHYSICAL PROPERTIES OF UNAGED ARCO AR4000,
AGED RESIDUE, AND ASTM D3381
SPECIFICATION LIMITS

<u>Property</u>	<u>Test Results</u>		<u>Specification Limits</u>
	<u>Unaged</u>	<u>RTFOT¹ Aged Residue</u>	
Penetration, 100g, 5 sec, 77F; 1/10 mm	78	48	25 min ²
Softening Point; °C	47.0	51.5	---
Absolute Viscosity, 60C, 30 cmHg; Poise	1612	3318	4000+1000 ²
Kinematic Viscosity, 135C; cSt	248	348	275 min ²
Ductility, 77F, 5cm/min; cm	---	7100	75 min ²
Flash Point, COC; °F	555	---	440 min
Solubility in Trichlor- ethylene; %	99.97	---	99.0 min
% Original Penetration, 77F	61		45 min
<hr/>			
Rolling Thin Film Oven Loss; %		0.09	

Notes: ¹Rolling Thin Film Oven Test
 ²Tests on RTFOT Residue

TABLE 6

PENETRATION, ABSOLUTE VISCOSITY, AND
KINEMATIC VISCOSITY OF AR1000, AR4000, AND
AR4000 WITH 2, 4, 8, 15, AND 25 PERCENT CALIFLUX

Property	AR1000	AR4000 + 25% Califlux	AR4000 + 15% Califlux	AR4000 + 8% Califlux	AR4000 + 4% Califlux	AR4000 + 2% Califlux	AR4000
Penetration, 100g, 5 sec, 77F; 1/10 mm	127	245	154	102	79	58	78
Absolute Viscosity, 30 cm Hg, 140F; Poise	662	268	495	838	1166	1484	1612
Kinematic Viscosity, 275F, cSt	178	109	150	196	229	248	248

- 3.2.6 Compositional analysis of the AR1000 and AR4000 unaged asphalt cements, Califlux GP and mixtures of the AR4000 and Califlux were performed by Sahuaro Petroleum using a modification of the Rostler-Sternberg Procedure (14). Test results are tabulated in Table 7.
- 3.3 Diluent. The diluent (Kerosene) used in this study was termed 410-H and was obtained from Chevron, USA in Phoenix, Arizona.

TABLE 7

ROSTLER COMPOSITIONAL ANALYSIS OF
CALIFLUX GP, ARL000, AR4000, AND AR4000
WITH 2, 6, AND 15 PERCENT CALIFLUX

Fraction	Califlux GP	% By Weight			
		AR1000	AR4000 + 15% Califlux	AR4000 + 6% Califlux	AR4000 + 2% Califlux
Asphaltenes, A	1.8	14.4	10.8	11.4	12.7
Nitrogen Bases, N	21.8	28.6	22.3	24.5	23.7
First Acidefins, A ₁	20.5	23.6	29.3	25.3	27.2
Second Acidefins, A ₂	32.8	21.6	25.6	26.4	25.1
Parafins, P	23.7	11.2	11.6	12.3	11.3

Rostler Parameter ¹	0.74	1.59	1.26	1.29	1.40	1.49
Gotolski Parameter ²	2.95	2.88	3.45	3.22	3.17	3.61

Note: ¹Rostler Parameter = $\frac{N + A_1}{P + A_2}$ ²Gotolski Parameter = $\frac{A_1 + A_2 + N}{A + P}$

4.0 EXPERIMENTAL DESIGNS AND DATA ANALYSIS

- 4.1 Experiments performed during this study were designed as either sequentially or completely randomized fixed factorial models with two replications per cell. Data were analyzed using conventional analysis of variance techniques. Results were tested for significance at the 95 and 99 percent confidence levels.
- 4.2 Prior to performing analysis of variance (ANOVA), homogeneity of variance was tested by the Foster and Burr q-test (15). Appropriate data transformations were used when necessary to comply with variance homogeneity constraints required for analysis of variance.
- 4.3 For several of the experiments, following ANOVA, significant effects were ranked using the Newman-Keuls multiple range test (16).
- 4.4 One, two, three, and four-way designs were used in the study. Details of the analytical models used for each experiment are contained in Volumes I through V of this report.

5.0 ASPHALT-RUBBER MIXTURE PREPARATION

5.1 Individual asphalt-rubber formulations were prepared in 1000 gm batches using the Arizona Torque-Fork mixer. Details on components of the Torque-Fork are contained in Appendix A. Upon completion of mixing, the asphalt-rubber mixture was separated into 8 ounce sealed tins and stored at 0F until tested. Material used for testing was reheated only once after mixing.

5.1.1 Asphalt-rubber mixtures used for both replications of a specific test were obtained from the same mixture batch. This procedure was used to eliminate the mixing variable in test results.

5.2 The mixing procedure used was as follows:

- A. Heat asphalt to 375F (191C) in the Torque-Fork mixing bowl with the stirring mechanism in operating at 200-300 rpm.
- B. Increase mixer speed to 500 rpm after the asphalt has reached 375F (191C).
- C. Introduce required amount of rubber into the asphalt as rapidly as possible while maintaining mixing speed at 500 rpm. The temperature will drop slightly, but do not adjust temperature controls.
- D. Remove mixed asphalt-rubber from Torque-Fork mixer, transfer to storage tins, and place material into freezer after material has reached room temperature.
- E. Record entire mixing cycle temperature and viscosity readings on a time chart.

5.3 To prepare a mixed asphalt-rubber mixture for testing, an 8 ounce tin of the specific mixture was removed from the freezer and heated to 275F on a hot-plate while constantly being stirred. When the material reached 275F, it was ready for specimen fabrication.

6.0 TESTING PROCEDURES

6.1 Vacuum Capillary Absolute Viscosity at 140F (60C).

6.1.1 The test method used was a modification of the standard method of test ASTM D2171-66 (13), as outlined by Green and Tolonen (1). Modifications to the procedure include:

- Use of large bore capillary viscometers (Asphalt Institute No. 800 for example) to accommodate swollen rubber particles.
- Reduction of applied vacuum from the standard 30 cm Hg to 10 cm to produce slower flow times and prevent separation of asphalt and rubber during capillary flow.

6.1.2 Use of the reduced vacuum required a correction of tube constants provided by the manufacturer. The correction is based on the following:

$$K = \frac{M_H}{(H-h)} \quad (1)$$

in which:

- K = Instrument constant in poises/seconds per cm Hg
- M_H = Multiplier constant for vacuum head of test (or calibration).
- H = Applied vacuum head (cm Hg).
- h = Average liquid head (cm Hg). Provided by viscometer manufacturer.

6.1.3 Viscosity is flow time in seconds multiplied by the constant M_H for the applied vacuum.

6.1.4 Viscometer tubes were loaded by means of a syringe consisting of a glass tube with a wood plunger. The syringe tube outside diameter is such that it loosely fits inside the viscometer tube. The wood plunger snugly fits inside the syringe tube. Procedure for filling the viscometer tube as described by Green and Tolonen (1) is as follows:

- 6.1.4.1 Viscometer, viscometer clamp stand, syringe tube, and plunger are preheated in an oven at 275F.
- 6.1.4.2 Asphalt-rubber is heated to 275F (135C) on a hotplate.
- 6.1.4.3 When the asphalt-rubber mixture reaches 275F, the syringe is loaded by suction from the wood plunger.
- 6.1.4.4 Transfer of asphalt-rubber from the syringe to the viscometer is by the plunger forcing material from the syringe tube while the tube is being withdrawn from the viscometer.
- 6.1.4.5 Immediately after filling, the viscometer is placed in the 140F bath. After 60 minutes in the bath, temperature equilibrium is assumed and the test is conducted.
- 6.1.5 Data obtained from this testing procedure for analysis is the absolute viscosity of the asphalt-rubber at 140F (60C).

6.2 Schweyer Rheometer

- 6.2.1 The Schweyer rheometer (17) is described as a constant stress rheometer that produces a rheogram of apparent viscosity (η) versus shear rate ($\dot{\gamma}$).
- 6.2.2 Principle of operation is that a sample of material is forced through a precision capillary by means of a constant load. Load is applied to the specimen by means of a plunger and downward movement of the plunger is monitored by a linear variable displacement transducer (LVDT).
- 6.2.3 Plunger movement is recorded as a function of time on a strip chart. The corrected plunger movement is equated to specimen flow through the capillary tube. Figure 4 shows a typical output curve.

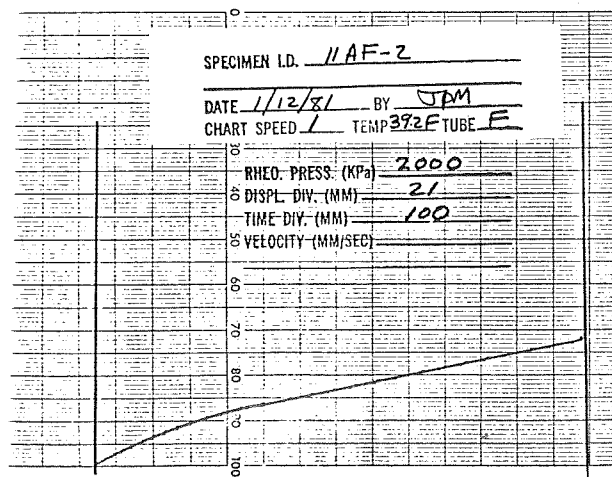
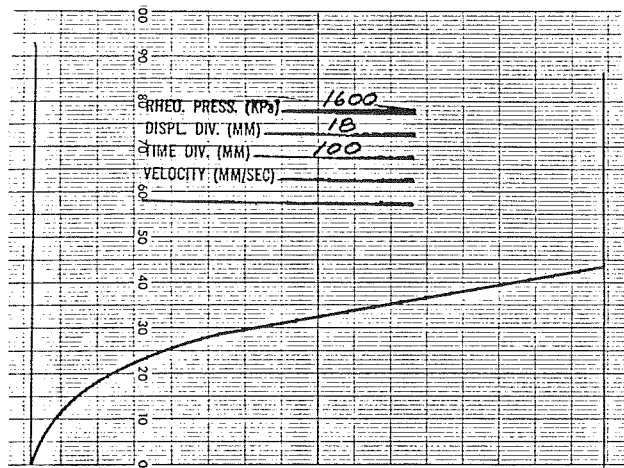
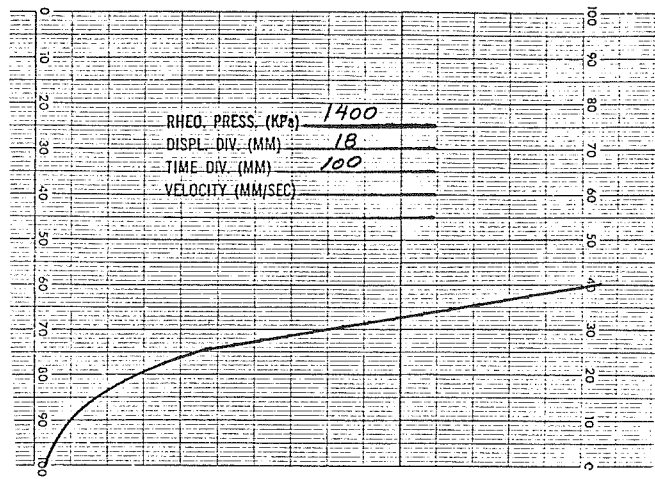


Figure 4 Schweyer Output Curve

6.2.4 Movement of the plunger in the initial stage is nonlinear, but becomes linear when equilibrium (constant) flow rate is established. Velocity measurements are made on the linear portion.

6.2.5 Test procedure and calculations are as follows:

6.2.5.1 Pressure on the loading ram is read directly from a gauge on the front of the instrument. Pressure is supplied by pressurized gaseous nitrogen and can be controlled to suit test conditions and material characteristics.

6.2.5.2 Applied force is the product of pressure (P) and the machine constant (M).

6.2.5.3 Shear stress (τ) is the product of force and and the tube constant (K_t).

$$\tau = (P \times M) (K_t) \quad (2)$$

6.2.5.4 Velocity (V) is the slope of the linear portion of the movement versus time tracing obtained from the strip chart recorder.

6.2.5.5 Shear rate ($\dot{\gamma}$) is calculated by multiplying velocity by a shear rate tube constant ($K_{\dot{\gamma}}$).

$$\dot{\gamma} = VK_{\dot{\gamma}} \quad (3)$$

6.2.5.6 Apparent viscosity (η_a) is given as:

$$\eta_a = \frac{\text{shear stress}}{\text{shear rate}} = \frac{\tau}{\dot{\gamma}}$$

and is expressed in Pascal-seconds (Pa-s)

6.2.5.7 After the test load is removed and the specimen returns to equilibrium, a different load is applied and a new apparent viscosity is calculated for the new shear stress and shear rate.

6.2.5.8 Several runs are made and a rheogram is developed that is a plot of log of apparent viscosity ($\log \eta_a$) versus log of shear rate ($\log \dot{\gamma}$) where each point on the curve represents one test run. These points form a straight line amenable to linear regression analysis and, theoretically, allow calculation of apparent viscosity for any shear rate. In this study, viscosities are reported for a 0.05 sec.^{-1} shear rate.

A typical rheogram and data used to generate it are shown in Figure 5.

6.2.6 Straight lines on a log log plot are represented by the power law equation:

$$y = a x^b \quad (5)$$

or for the rheogram:

$$\eta_a = a(\dot{\gamma})^b \quad (6)$$

where b is the slope of the straight line.

6.2.6.1 Materials with horizontal rheogram plots ($b = 0$) are Newtonian fluids and are not shear susceptible. In other words apparent viscosity is constant over a range of shear rates.

6.2.6.2 Materials with slopes down to the right ($b < 0$) are termed pseudoplastic. These materials are shear susceptible: as shear rate increases viscosity decreases.

6.2.6.3 Materials with slopes up to the right ($b > 0$) are termed dilatant. Shear susceptibility of these materials is exhibited by increased viscosity with increased shear rate. This property may be of interest in the asphalt-rubber field since observations have been reported that performance in runway touchdown areas (high shear rate) is better than in areas where traffic is relatively slow moving. If the observations are valid, this performance is the opposite of what would be expected from pseudoplastic, and probably, Newtonian materials.

IDENTIFICATION 2BA1

CHART NO. 2 TEMP. 40 TUBE F

(L), LVDT Chart Dsp., mm	24	14	11	6		
(T), Time Chart Dsp., mm	100	100	100	100		
(CS), Chart Speed, cm/min	1	1	1	1		
(P), Pressure, KPa	2000	1600	1200	800		
τ Pa	1.8×10^6	1.4×10^6	1.1×10^6	7.0×10^5		
V mm/sec	3.2×10^{-4}	1.4×10^{-4}	1.5×10^{-4}	8.0×10^{-5}		
$\dot{\gamma}$ sec ⁻¹	2.4×10^{-3}	1.4×10^{-3}	1.1×10^{-3}	6.0×10^{-4}		
η_a Pa-sec	7.3×10^{-8}	1.0×10^{-7}	9.6×10^{-8}	1.2×10^{-7}		

Regression ($\eta_a = a\dot{\gamma}^b$): $a = \frac{1.1 \times 10^{-8}}{}$ $b = \frac{-3.2 \times 10^{-1}}{}$ $R^2 = \frac{0.89}{}$
 Rheologic Constants : $\eta_{0.05} = \frac{2.9 \times 10^{-8}}{}$ $\eta_{1.0} = \frac{1.1 \times 10^{-8}}{}$ $c = \frac{0.68}{}$

Plotting Points:

$\dot{\gamma}$	5.0×10^{-3}	5.0×10^{-4}	
η_a	6.1×10^{-8}	1.3×10^{-7}	

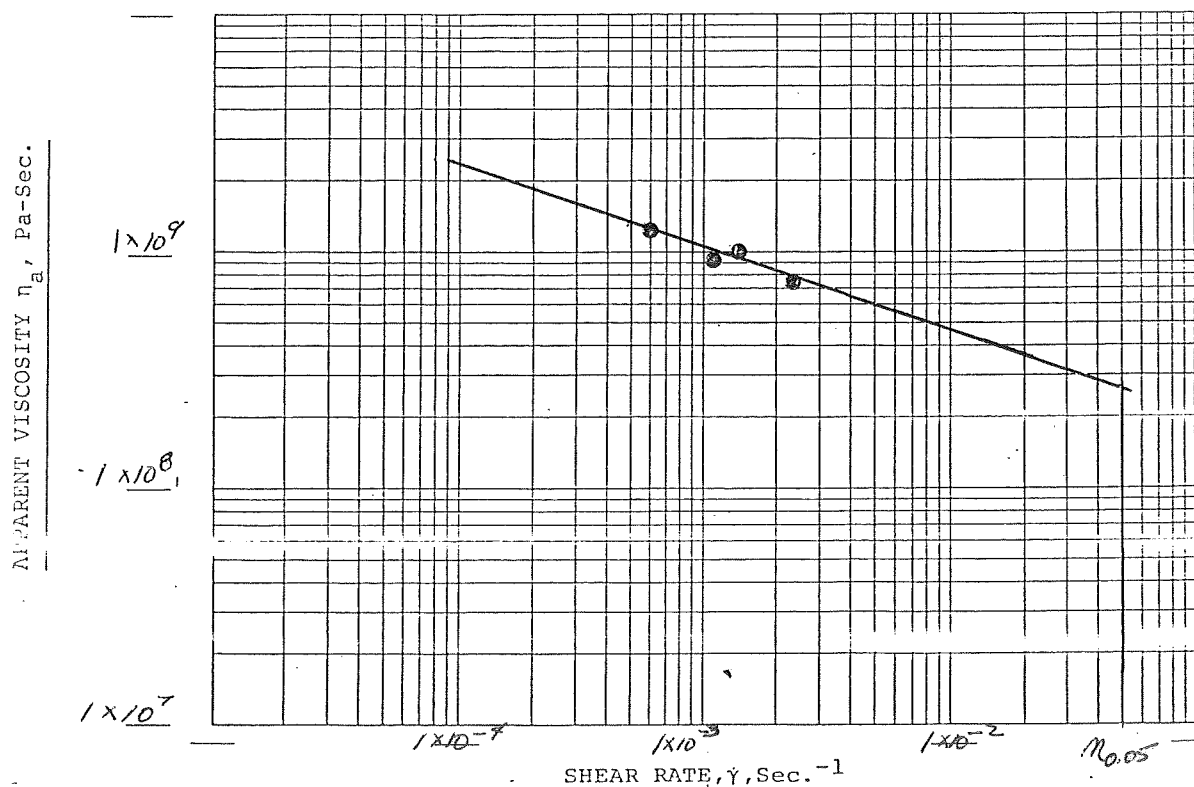


Figure 5 Schweyer Rheogram

6.2.7 Shear susceptibility is a function of the slope of the rheogram. Newtonian materials have zero slope, pseudoplastic materials have negative slopes, and dilatant materials have positive slopes. The rheogram is described by a characteristic apparent viscosity at a given shear rate and the power law parameters a and b (slope).

6.2.7.1 Shear susceptibility in this study is reported by the parameter C . The shear susceptibility index is defined as:

$$C = 1 + \text{slope} = 1 + b \quad (7)$$

6.2.7.2 Newtonian materials ($b = 0$) have a shear susceptibility index of 1.

6.2.7.3 Pseudoplastic materials ($b < 0$) have shear susceptibility indices of less than 1.

6.2.7.4 Dilatant materials ($b > 0$) have shear susceptibility indices greater than 1.

6.2.8 Specimen tubes used for the constant stress rheometer consist of a specimen or flow tube of 9.47 mm (approximately 3/8 inches) in diameter with a capillary threaded onto the end of the flow tube which is smaller in diameter than the flow tube. Selection of capillary size is based on relative viscosity of the material under test conditions. Low viscosity materials require smaller capillaries than high viscosity materials.

6.2.8.1 The pressurized ram or plunger forces the large plug of material being tested down the flow tube and through the capillary. The capillary dictates flow rate and hence shear rate and shear stress. There is some question as to size effects of the capillary on measured viscosity. It should be expected that, for a given material under constant environmental conditions, different tube sizes would give the same viscosity if capillary size effects were not present. For asphalt-rubber, the possibility of particle interference needs to be considered as rubber particles may clog capillary openings and interfere with flow. If interference exists, measurements could be misleading since, instead of representing asphalt-rubber mass viscosity, elasticity of rubber or flow of asphalt around rubber particles could be measured.

6.2.9 Schwyer Rheometer tests at -20F (-29C), 39.2F (4C), and 77F (25C) were performed in this study using two different capillary tube sizes -"F" (4.650 mm diameter) and "G" (9.700 mm diameter). Apparent viscosity at a shear rate of 0.05 sec⁻¹ and shear susceptibility constants C were calculated and analyzed using both F and G tubes.

6.3 Force-Ductility

6.3.1 The force-ductility test used during this project is a modification of the standard ductility test (ASTM D113-77, "Ductility of Bituminous Materials") and is similar to the procedure developed by Anderson and Wiley (18). Basically, the test is described as follows:

6.3.1.1 A modified ductility briquet is stretched at a constant rate in a constant temperature bath. The load required to stretch the specimen is continuously monitored with a load cell connected in series with the test specimen. Specimen elongation is also monitored during the test.

6.3.1.2 Asphalt-rubber test specimens are cast in a modified ductility mold as shown in Figure 6. This mold geometry provides a constant cross section so that the compliance of the material being tested can be evaluated.

6.3.1.3 Testing was performed at -20F (-29C), 39.2F (4C), and 77F (25C) using an extension rate of 1 cm per minute.

6.3.2 During the test, the following measurements are taken:

6.3.2.1 Initial gauge length (approximately 50 mm) between two gauge marks placed at each end of the specimen constant cross section portion.

6.3.2.2 Load obtained from the load cell output at 2 minute intervals.

6.3.2.3 Length between gauge marks at the same time load readings are taken.

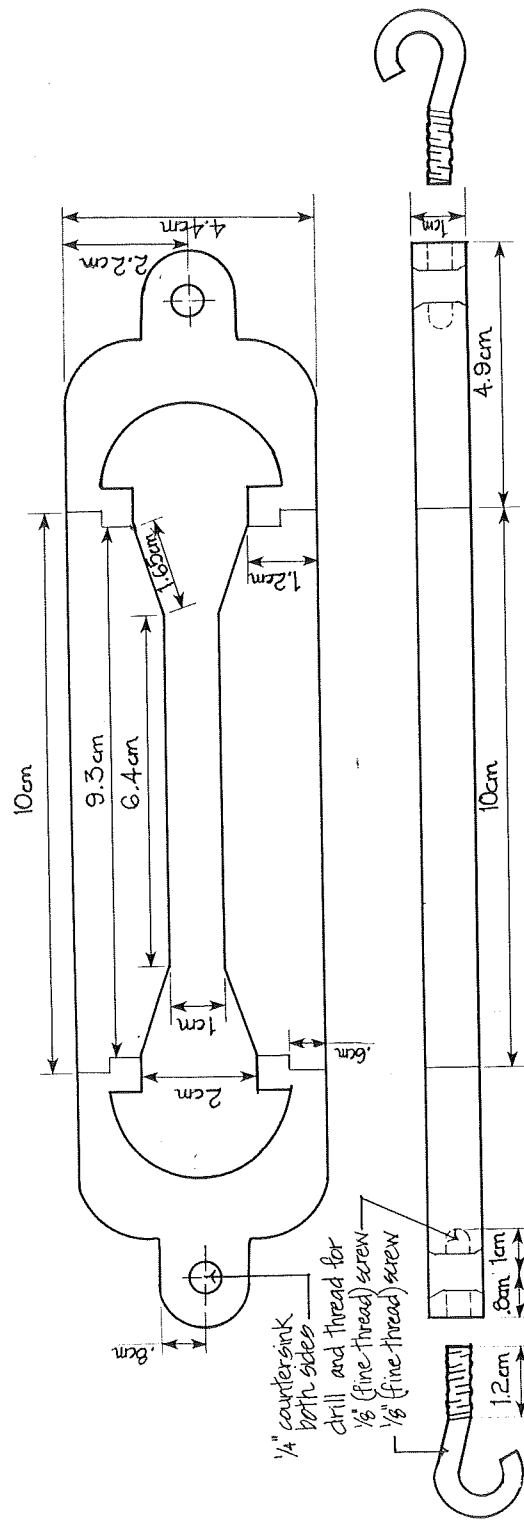


Figure 6 Modified Ductility Mold

6.3.3 From the above measurements, ten parameters are calculated and reported.

6.3.3.1 Load at Failure - reported in pounds.

6.3.3.2 Elongation at Failure - calculated as the length between gauge marks at failure minus initial gauge length in millimeters.

6.3.3.3 Engineering Stress at Failure - calculated as the load at failure per unit of original specimen cross section as follows:

$$\sigma_e = \frac{P}{A} \quad (8)$$

in which:

σ_e = Engineering stress at failure, psi
P = Load at Failure, pounds
A = Original specimen cross section, in²

6.3.3.4 Engineering Strain at Failure - calculated as follows:

$$\epsilon_e = \frac{F - I}{F} \quad (9)$$

in which:

ϵ_e = Engineering strain at failure, mm/mm
F = Gauge length at failure, mm
I = Initial gauge length, mm

6.3.3.5 True Stress at Failure - based on the specimen cross section at failure calculated assuming a constant specimen volume between gauge marks. True stress at failure was calculated using the following formula:

$$\sigma_t = \frac{P}{A_c} \quad (10)$$

in which:

σ_t = True stress at failure, psi
P = Load at failure, pounds
 A_c = Calculated specimen cross sectional area, in²

6.3.3.6 True Strain at Failure - calculated as:

$$\epsilon_t = (1 + \epsilon_e) \quad (11)$$

in which:

ϵ_t = True strain at failure, mm/mm

ϵ_e = Engineering strain at failure, mm/mm

6.3.3.7 Engineering Creep Compliance at Failure - calculated as engineering strain at failure divided by engineering stress at failure. Units are psi⁻¹.

6.3.3.8 True Creep Compliance at Failure - calculated as true strain at failure divided by true stress at failure. Units are psi⁻¹.

6.3.3.9 Maximum True Creep Compliance - determined as the maximum true creep compliance value reached throughout the test duration.

6.3.3.10 Time to Maximum True Creep Compliance - the elapsed time in minutes to reach maximum true creep compliance.

6.4 Sliding Plate Microviscometer

6.4.1 Eight ounce portions of asphalt-rubber mixtures prepared at Western Technologies, Inc. during this study were shipped to Petroleum Sciences, Inc., Spokane, Washington for testing using the sliding plate microviscometer at 32F (0C).

6.4.2 The testing procedure used is a modification of the elastic rebound procedure described by Green and Tolonen (1). The procedure used is discussed in following sections.

6.4.2.1 Apparatus consists of:

- sliding plate microviscometer as designed by Shell Development Company as described by Fenijn (19).

- glass microviscosity plates 20 mm x 30 mm x 10 mm.

- spacers 2.54 mm in thickness.
 - temperature control bath
- 6.4.2.2 A 20 x 25 mm specimen of asphalt-rubber which is 2.5 mm in thickness is pour formed between the glass plates.
- 6.4.2.3 The testing procedure used consists of:
- Placing the prepared specimen in the sliding plate microviscometer and lowering the specimen into a constant temperature bath until thermal equilibrium is reached at the desired testing temperature.
 - When temperature equilibrium is reached, a 500 gram shearing load is applied to the plates for 30 minutes and resulting deflections monitored.
 - During this 30 minute creep cycle, 22 deflection measurements are obtained as a function of time.
 - At the end of the 30 minute creep cycle, the 500 gram shear load is removed permitting the specimen to rebound under a no shear stress condition. Resulting rebound or recovery is monitored for 30 minutes. Again, 22 deflection measurements are obtained as a function of time. This procedure completes first cycle testing.
 - Following completion of the 30 minute recovery phase, the specimen is once again loaded with the 500 gram load and allowed to creep for 30 minutes. Deflection after 30 minutes of creep is recorded and the 500 gram load then removed and the specimen permitted to recover.
 - At the end of 30 minute recovery period, deflection is recorded and the specimen allowed to recover for 20 hours. At the end of the 20 hour recovery period, the total deflection is measured. This completes the second cycle and is the end of the test.

6.4.3 The response of the samples to shear stress was separated into a non-recoverable apparent viscosity component and a recoverable viscoelastic component. A schematic diagram of the strain vs time curve for the entire test is shown in Figure 7. Apparent viscosity was calculated from the non-recoverable strain (after conditioning for 20 hours following completion of the test), and the 9807 Pa stress (500 gm shear load) which is applied to the sample for 60 minutes as follows:

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (12)$$

in which:

η = apparent viscosity
 τ = applied shear stress (9807 Pa)
 $\dot{\gamma}$ = shear strain rate calculated based on the measured non-recoverable flow, specimen thickness, and 3600 second loading time.

The viscoelastic creep component of the data was calculated by subtracting the strain resulting from viscous flow from the total strain at each time interval. Viscous effects are not present in the strain recovery portion of the test, thus the data, as measured, is representative of the viscoelastic strain recovery response.

The viscoelastic portions of the creep and strain recovery data were each fitted to the following mathematical model as proposed by Green and Tolonen (1):

$$S_v = S_m(1 - e^{-bt^n}) \quad (13)$$

in which:

S_v = elastic strain at any time interval
 t = time
 S_m = maximum elastic strain obtainable at $t = \infty$
 b, n = constants

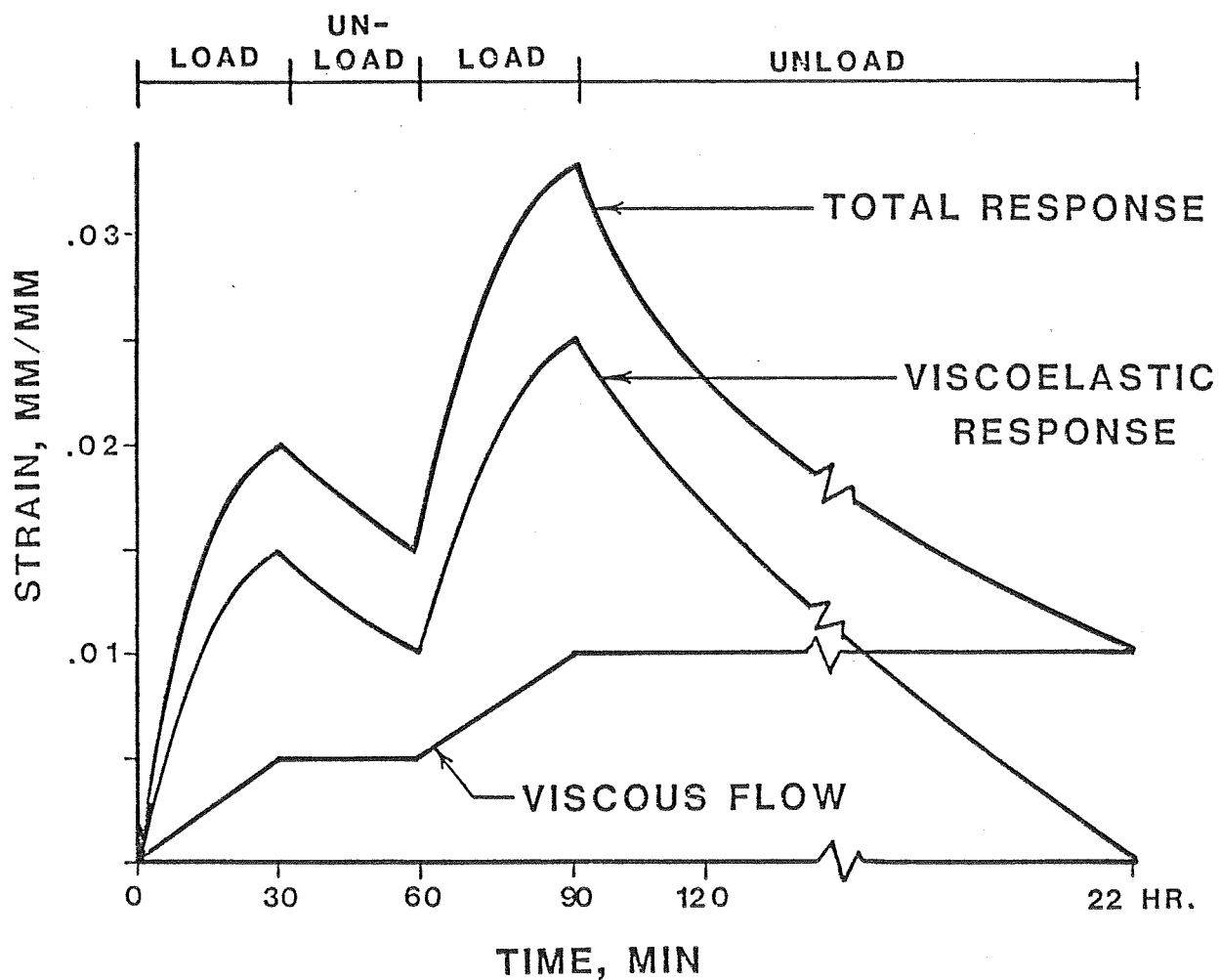


Figure 7 Strain as a Function of Time During the Creep-Strain Recovery Test

Equation 13 is not theoretically derived, but permits separation of viscous and elastic responses. In equation 13, S_m indicates the degree of elasticity of the asphalt-rubber while b and n are related to viscous characteristics.

- 6.4.4 For calculation purposes, equation 13 was rearranged to:

$$\frac{S_m}{S_m - S_v} = e b t^n \quad (14)$$

The natural logarithm of the natural logarithm of equation 13 is then taken to yield:

$$\ln \ln \frac{S_m}{S_m - S_v} = n \ln t + \ln b \quad (15)$$

Values for S_m were inserted into equation 15 on a trial and error basis and results compared to measured values until a maximum correlation coefficient was obtained. Correlation coefficients (r^2) were generally above 0.99. Coefficients b and n were determined as the intercept and slope of the $\ln \ln \frac{S_m}{S_m - S_v}$ vs. $\ln t$ plot (Figure 8) using the S_m value which gave the maximum correlation coefficient.

- 6.4.5 Parameters obtained from sliding plate microviscometer testing which are reported, analyzed and discussed are:

- Apparent viscosity
- First cycle 30 minute creep in microns.
- First cycle 30 minute recovery in microns.
- Second cycle 30 minute creep in microns.
- Second cycle 30 minute recovery in microns.
- Second cycle 20 hour recovery in microns.
- Second cycle 20 hour recovery minus second cycle 30 minute recovery in microns.

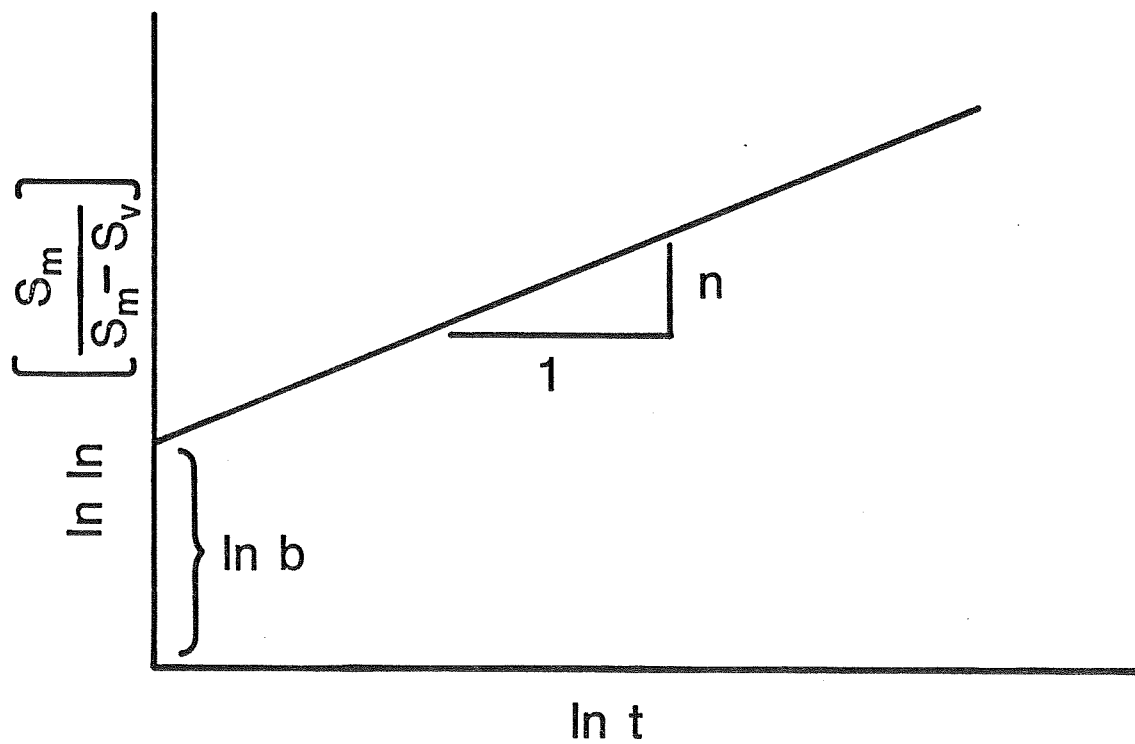


Figure 8 $\ln \ln \left(\frac{S_m}{S_m - S_v} \right)$ vs. $\ln t$ Plot

- Percent first cycle recovery calculated as:

$$\frac{\text{First cycle recovery (30 min)}}{\text{First cycle creep (30 min)}} \times 100\%$$

- First cycle creep rheological constant S_m
- First cycle creep rheological constant b
- First cycle creep rheological constant n
- First cycle recovery rheological constant S_m
- First cycle recovery rheological constant b
- First cycle recovery rheological constant n

6.5 Viscosity During Mixing at 375F (191C) by the Arizona Torque Fork.

6.5.1 Viscosity during mixing at 375F (191C) by the Torque Fork was determined by correlation of millivolt readings from the servodyne unit with calibration readings obtained for S30000 Calibration oil obtained from Cannon Instrument Company. Details of construction and calibration of the Torque-Fork are contained in Appendix A.

6.5.2 During mixing Servodyne millivolt output readings were taken at 15 minutes and 1 hour following reestablishment of mixing temperature (375F) after introduction of rubber. Millivolt readings were transformed to viscosity in poise using techniques described in Appendix A..

6.6 Viscosity During Mixing at 375F (191C) by the Haake Rotational Viscometer.

6.6.1 Viscosity during mixing at 375F (191C) was determined at 15 minutes and 1 hour following reestablishment of mixing temperature (375F) after introduction of rubber by reading directly from the Haake viscometer. Viscosity readings are reported in poise.

- 6.7 Low temperature fracture tests were performed using the McDonald procedure, Method B as described in Appendix B.
- 6.8 Ring and ball softening point was determined in accordance with ASTM D2398-76, "Softening Point of Bitumen in Ethylene Glycol (Ring-and-Ball)" (13). ASTM D36-76 was not used due to the high softening point of several of the asphalt-rubber mixtures tested.